

## SEARCH REQUEST FORM

Scientific and Technical Information Center

Requester's Full Name: Reyes, Hector Examiner #: 70264 Date: 5/11/98Art Unit: 1625 Phone Number: 301 405-1153 Serial Number: 101070365Mail Box and Bldg/Room Location: \_\_\_\_\_ Results Format Preferred (circle): PAPER DISK E-MAIL

3001-24A16  
 If more than one search is submitted, please prioritize searches in order of need.

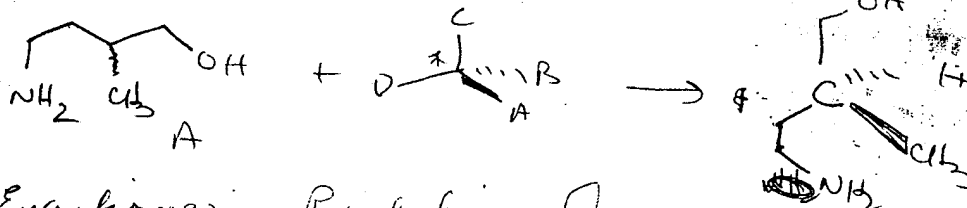
\*\*\*\*\*  
 Please provide a detailed statement of the search topic, and describe as specifically as possible the subject matter to be searched. Include the elected species or structures, keywords, synonyms, acronyms, and registry numbers, and combine with the concept or utility of the invention. Define any terms that may have a special meaning. Give examples or relevant citations, authors, etc, if known. Please attach a copy of the cover sheet, pertinent claims, and abstract.

Title of Invention: See copy of Bifra

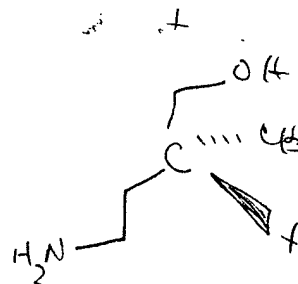
Inventors (please provide full names): \_\_\_\_\_

Earliest Priority Filing Date: \_\_\_\_\_

\*For Sequence Searches Only\* Please include all pertinent information (parent, child, divisional, or issued patent numbers) along with the appropriate serial number.



Enantiomeric Resolution of  
 A using any possible  
 optical agent and  
 specially those disclose  
 in claims.



POINT OF CONTACT:  
 PAUL SCHULWITZ  
 TECHNICAL INFO. SPECIALIST  
 CM1 6B06 TEL. (703) 305-1954

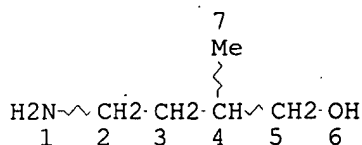
Thanks

## STAFF USE ONLY

Type of Search	Vendors and cost where applicable
Searcher: _____	NA Sequence (#) _____ STN <u>324.47</u>
Searcher Phone #: _____	AA Sequence (#) _____ Dialog _____
Searcher Location: _____	Structure (#) <u>2</u> Questel/Orbit _____
Date Searcher Picked Up: <u>5/2</u>	Bibliographic _____ Dr. Link _____
Date Completed: <u>5/2</u>	Litigation _____ Lexis/Nexis _____
Searcher Prep & Review Time: <u>15</u>	Fulltext _____ Sequence Systems _____
Clerical Prep Time: _____	Patent Family _____ WWW/Internet _____
Online Time: <u>12</u>	Other _____ Other (specify) _____

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## NODE ATTRIBUTES:

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

## GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED

NUMBER OF NODES IS 7

## STEREO ATTRIBUTES: NONE

L3 52640 SEA FILE=REGISTRY ABB=ON PLU=ON NC=1 AND C=5 NOT RSD/FA  
 L4 4 SEA FILE=REGISTRY SUB=L3 SSS FUL L1  
 L5 18 SEA FILE=HCAPLUS ABB=ON PLU=ON L4(L) PREP/RL  
 L6 16 SEA FILE=HCAPLUS ABB=ON PLU=ON L4(L) (RACT OR RCT OR RGT)/RL  
 L7 9 SEA FILE=HCAPLUS ABB=ON PLU=ON L5 AND L6  
 L8 8 SEA FILE=HCAPLUS ABB=ON PLU=ON L7 AND (STEREO? OR ENANT? OR  
 OPTICAL?)  
 L9 9 SEA FILE=HCAPLUS ABB=ON PLU=ON L7 OR L8

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L9 ANSWER 1 OF 9 HCAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2003:298987 HCAPLUS

TITLE: Preparation and purification of **optically**  
 active 4-amino-2-methylbutan-1-ol and its sulfonic  
 acid salts

INVENTOR(S): Sakano, Kunihiro

PATENT ASSIGNEE(S): Mitsubishi Rayon Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

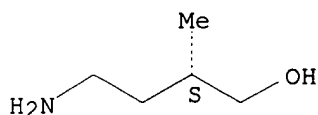
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2003113146	A2	20030418	JP 2001-307708	20011003
PRIORITY APPLN. INFO.:			JP 2001-307708	20011003

AB **Optically** active 4-amino-2-methylbutan-1-ol is prepd. by  
 recrystn. of RSO<sub>3</sub><sup>-</sup> +NH<sub>3</sub>CH<sub>2</sub>CH<sub>2</sub>CHMeCH<sub>2</sub>OH [R = C1-20 (un)substituted alkyl,  
 alkoxy, acyloxy, (un)substituted amino, Ph, naphthyl] and treatment with  
 alkalies in solvents. (S)-4-amino-2-methylbutan-1-ol was treated with  
 MeSO<sub>3</sub>H in acetonitrile at 60.degree. and recrystd. to give a salt with  
 95%e.e.

IC ICM C07C213-10  
 ICS C07C215-08

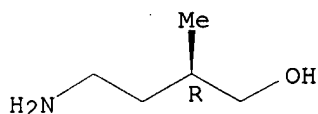
CC 23-7 (Aliphatic Compounds)  
 IT INDEXING IN PROGRESS  
 IT **44565-28-8P**, (S)-4-Amino-2-methylbutan-1-ol **88390-32-3P**  
 RL: PUR (Purification or recovery); **RCT (Reactant)**; **PREP**  
**(Preparation); RACT (Reactant or reagent)**  
 (purifn. of **optically** active aminomethylbutanol by reaction  
 with sulfonic acids and recrystn.)  
 IT **44565-28-8P**, (S)-4-Amino-2-methylbutan-1-ol **88390-32-3P**  
 RL: PUR (Purification or recovery); **RCT (Reactant)**; **PREP**  
**(Preparation); RACT (Reactant or reagent)**  
 (purifn. of **optically** active aminomethylbutanol by reaction  
 with sulfonic acids and recrystn.)  
 RN 44565-28-8 HCAPLUS  
 CN 1-Butanol, 4-amino-2-methyl-, (2S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



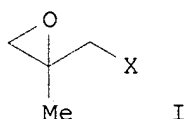
RN 88390-32-3 HCAPLUS  
 CN 1-Butanol, 4-amino-2-methyl-, (2R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L9 ANSWER 2 OF 9 HCAPLUS COPYRIGHT 2003 ACS  
 ACCESSION NUMBER: 2003:196444 HCAPLUS  
 DOCUMENT NUMBER: 138:221235  
 TITLE: Preparation of **optically** active  
 3-methyl-4-hydroxybutylamines  
 INVENTOR(S): Watanabe, Hisayuki; Kawamura, Yasuo; Hashiba, Isao  
 PATENT ASSIGNEE(S): Nissan Chemical Industries, Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 9 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2003073344	A2	20030312	JP 2001-262569	20010831
PRIORITY APPLN. INFO.:			JP 2001-262569	20010831
OTHER SOURCE(S):		MARPAT 138:221235		
GI				



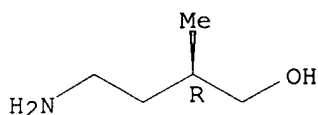
- AB. **Optically** active OH-protected 3-methyl-4-hydroxybutylamines are prep'd. by reaction of oxiranes I [X = halo, halo-(un)substituted C1-4 alkylsulfonyloxy, arylsulfonyloxy] with metal cyanides, asym. hydrogenation of 3-cyano-2-methyl-2-propenol, redn. of **optically** active 2-methyl-3-hydroxybutyronitrile, and protection of OH group of **optically** active 3-methyl-4-hydroxy butylamine. The compds. are useful as intermediate for (9R)-7-[3,5-bis(trifluoromethyl)benzyl]-6,7,8,9,10,11-hexahydro-5-(4-hydroxymethylphenyl)-9-methyl-6,13-dioxo-13H-[1,4]-diazocino[2,1-g][1,7]naphthyridine (no data). 2-Chloromethyl-2-methyloxirane was reacted with NaCN in H<sub>2</sub>O at room temp. overnight, which was hydrogenated in MeOH in the presence of [Rh(nbd)(S,S)-tert-Bu-BisP\*]BF<sub>4</sub> under 0.7 MPa H for 3 days, and reduced with LiAlH<sub>4</sub> at room temp. for 1 h to give (R)-3-methyl-4-hydroxybutylamine, which was reacted with 3,4-dihydropyran in the presence of HCl in THF at room temp. for 5 h to give 80% (R)-3-methyl-4-(2-tetrahydropyranyloxy)butylamine.
- IC ICM C07C213-02  
ICS C07B053-00; C07C215-08; C07C253-30; C07C255-12; C07C255-14;  
C07D309-12; C07B061-00; C07M007-00
- CC 23-7 (Aliphatic Compounds)  
Section cross-reference(s): 28
- IT 98-88-4, Benzoyl chloride 110-87-2  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(for protecting group; prep'n. of **optically** active methylhydroxybutylamines by reaction of methyloxirane with metal cyanides, hydrogenation, and redn.)
- IT **88390-32-3P**  
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(in protection; prep'n. of **optically** active methylhydroxybutylamines by reaction of methyloxirane with metal cyanides, hydrogenation, and redn.)
- IT 52900-11-5P 217189-83-8P 501108-20-9P, 3-Benzoyloxy-2-methyl-1-cyano-1-propene  
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(prep'n. of **optically** active methylhydroxybutylamines by reaction of methyloxirane with metal cyanides, hydrogenation, and redn.)
- IT 183551-55-5P 258265-78-0P, (S)-3-Methyl-4-benzyloxybutyronitrile  
RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)  
(prep'n. of **optically** active methylhydroxybutylamines by reaction of methyloxirane with metal cyanides, hydrogenation, and redn.)
- IT 207606-22-2P  
RL: PNU (Preparation, unclassified); PREP (Preparation)  
(prep'n. of **optically** active methylhydroxybutylamines by reaction of methyloxirane with metal cyanides, hydrogenation, and redn.)

IT 598-09-4, 2-Chloromethyl-2-methyloxirane  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (prepn. of **optically** active methylhydroxybutylamines by  
 reaction of methyloxirane with metal cyanides, hydrogenation, and  
 redn.)

IT 88390-32-3P  
 RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic  
 preparation); PREP (Preparation); RACT (Reactant or  
 reagent)  
 (in protection; prepn. of **optically** active  
 methylhydroxybutylamines by reaction of methyloxirane with metal  
 cyanides, hydrogenation, and redn.)

RN 88390-32-3 HCAPLUS  
 CN 1-Butanol, 4-amino-2-methyl-, (2R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L9 ANSWER 3 OF 9 HCAPLUS COPYRIGHT 2003 ACS  
 ACCESSION NUMBER: 2002:886132 HCAPLUS  
 DOCUMENT NUMBER: 137:384569  
 TITLE: Purification of (2R)-4-amino-2-methylbutan-1-ol  
 INVENTOR(S): Nohira, Hiroyuki; Watanabe, Hisayuki; Shiratori,  
 Motohito; Seki, Kenichi; Kawamura, Yasuo; Hashiba,  
 Isao  
 PATENT ASSIGNEE(S): Nissan Chemical Industries, Ltd., Japan  
 SOURCE: Jpn. Kokai Tokkyo Koho, 3 pp.  
 CODEN: JKXXAF  
 DOCUMENT TYPE: Patent  
 LANGUAGE: Japanese  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2002332263	A2	20021122	JP 2001-137458	20010508

PRIORITY APPLN. INFO.: JP 2001-137458 20010508

AB The compd. (I) is purified by crystn. of I terephthalic acid salt and  
 treatment of the resulting salts with bases. I was treated with  
 terephthalic acid in EtOH at 80.degree., left overnight, and treated with  
 NaOH in H2O to give I with 99.1% purity.

IC ICM C07C209-88  
 ICS C07C215-08; C07B057-00; C07M007-00

CC 23-7 (Aliphatic Compounds)

ST aminomethylbutanol **optical** resoln terephthalic acid

IT 88390-32-3P  
 RL: PUR (Purification or recovery); RCT (Reactant); SPN  
 (Synthetic preparation); PREP (Preparation); RACT (Reactant  
 or reagent)  
 (purifn. of 4-amino-2-methylbutan-1-ol)

IT 88390-32-3P

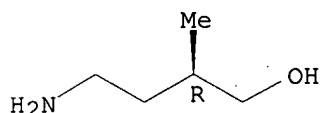
RL: PUR (Purification or recovery); RCT (Reactant); SPN  
(Synthetic preparation); PREP (Preparation); RACT (Reactant  
or reagent)

(purifn. of 4-amino-2-methylbutan-1-ol)

RN 88390-32-3 HCAPLUS

CN 1-Butanol, 4-amino-2-methyl-, (2R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L9 ANSWER 4 OF 9 HCAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2002:807297 HCAPLUS

DOCUMENT NUMBER: 137:325150

TITLE: Preparation of **optically** active

3-substituted 4-hydroxybutylamines

INVENTOR(S): Watanabe, Hisayuki; Kamisaka, Hiroyuki; Oda, Takashi;  
Seki, Kenichi; Kawamura, Yasuo

PATENT ASSIGNEE(S): Nissan Chemical Industries, Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 18 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2002308838	A2	20021023	JP 2001-299919	20010928
PRIORITY APPLN. INFO.:		JP 2001-31655	A	20010208
OTHER SOURCE(S): MARPAT 137:325150				

AB **Optically** active H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CHR<sub>1</sub>CH<sub>2</sub>OR<sub>4</sub> [I; R<sub>1</sub> = C<sub>1</sub>-6 (cyclo)alkyl, C<sub>2</sub>-6 (cyclo)alkenyl, C<sub>2</sub>-6 (cyclo)alkynyl, Ph, benzyl; R<sub>4</sub> = protective group], useful as intermediates for a dysuria treating agent, etc., are prep'd. by redn. of **optically** active NCCH<sub>2</sub>CHR<sub>1</sub>CO<sub>2</sub>R<sub>2</sub> (II; R<sub>1</sub> = same as I; R<sub>2</sub> = H, alkali metal, alk. earth metal, any group given for R<sub>1</sub>) and OH protection. (R)-II (R<sub>1</sub> = Me, R<sub>2</sub> = Et) (prepn. given) was reduced by LiAlH<sub>4</sub> in THF at 0.degree. for 1 h and treated with aq. NaOH to give 84% (R)-I (R<sub>1</sub> = Me, R<sub>4</sub> = H), which was treated with 3,4-dihydropyran in THF in the presence of HCl at 0.degree. for 5 h to give 80% (R)-I (R<sub>1</sub> = Me, R<sub>2</sub> = 2-tetrahydropyranyl).

IC ICM C07C213-02  
ICS C07C215-08; C07C253-30; C07C255-19; C07C255-22; C07D309-12;  
A61K031-4375; A61P013-02; C07B053-00; C07B061-00; C07D487-04;  
C07M007-00

CC 23-19 (Aliphatic Compounds)

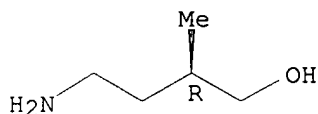
ST hydroxybutylamine **optically** active prepn cyanopropionate redn

IT 88390-32-3P 110171-23-8P 167778-98-5P 217189-83-8P  
454685-94-0P 454685-96-2P 454685-97-3P 473445-13-5P 473445-14-6P

RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic  
preparation); PREP (Preparation); RACT (Reactant or  
reagent)

(prepn. of **optically** active hydroxybutylamines)  
 IT 183551-55-5P  
 RL: IMF (Industrial manufacture); SPN (Synthetic preparation); PREP (Preparation)  
 (prepn. of **optically** active hydroxybutylamines)  
 IT 105-34-0, Methyl cyanoacetate 1116-98-9, tert-Butyl cyanoacetate 13361-32-5, Allyl cyanoacetate 42411-39-2 74497-15-7  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (prepn. of **optically** active hydroxybutylamines)  
 IT 88390-32-3P  
 RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (prepn. of **optically** active hydroxybutylamines)  
 RN 88390-32-3 HCAPLUS  
 CN 1-Butanol, 4-amino-2-methyl-, (2R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L9 ANSWER 5 OF 9 HCAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2002:205059 HCAPLUS

DOCUMENT NUMBER: 136:247347

TITLE: Recovery of **optically** active organic acid in preparation of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resolution

INVENTOR(S): Yamaguchi, Yasumasa; Hamanaka, Koji; Inaba, Hideyuki

PATENT ASSIGNEE(S): Mitsubishi Rayon Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 7 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2002080434	A2	20020319	JP 2000-268506	20000905
PRIORITY APPLN. INFO.:			JP 2000-268506	20000905
OTHER SOURCE(S): MARPAT 136:247347				

AB (R)- or (S)-4-amino-2-methylbutan-1-ol [(R)- or (S)-I] is prepd. by contacting a soln. contg. (R)- or (S)-I and **optically** active org. acid, with alkali for salt decompn. and filtering. (R)- or (S)-I is recovered from the filtrate and the resulting solid org. acid salt is collected by the solid-liq. sepn. The collected salt is contacted with acid in solvent and crystd. to recover the **optically** active org. acid. Thus, (RS)-I was treated with (R)-2-chloromandelic acid (II) in EtOH under heating, crystd., and filtered to give a soln. contg. (R)-I, (S)-I, and II, which was treated with EtONa and filtered to give II salt and filtrate. The collected salt was dissolved in hot water, treated with

*n Part*

- 70% H<sub>2</sub>SO<sub>4</sub>, and cooled to recover 87% II. The filtrate was distd. to give (S)-I with 61% e.e. with 88% recovery.
- IC ICM C07C213-10  
ICS C07C215-08; C07B057-00; C07M007-00
- CC 23-7 (Aliphatic Compounds)
- ST **optically** active chloromandelic acid recovery aminomethylbutanol resolu
- IT Carboxylic acids, preparation  
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(chiral; recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resolu.)
- IT Sulfonic acids, preparation  
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(**optically** active; recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resolu.)
- IT Resolution (separation)  
(recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resolu.)
- IT Alcohols, uses  
RL: NUU (Other use, unclassified); USES (Uses)  
(recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resolu.)
- IT Alkali metal hydroxides  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resolu.)
- IT Metal alkoxides  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resolu.)
- IT **44565-28-8P**, (S)-4-Amino-2-methylbutan-1-ol  
RL: IMF (Industrial manufacture); PUR (Purification or recovery); SPN (Synthetic preparation); **PREP (Preparation)**  
(recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resolu.)
- IT 52950-18-2P  
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resolu.)
- IT 64-17-5, Ethanol, uses 67-56-1, Methanol, uses 67-63-0, Isopropyl alcohol, uses 71-36-3, Butanol, uses 7732-18-5, Water, uses  
RL: NUU (Other use, unclassified); USES (Uses)  
(recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resolu.)
- IT 124-41-4, Sodium methylate 141-52-6, Sodium ethylate 1310-73-2, Sodium



hydroxide, reactions 44565-27-7, 4-Amino-2-methylbutan-1-ol

RL: RCT (Reactant); RACT (Reactant or reagent)

(recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resln.)

IT 44565-28-8P, (S)-4-Amino-2-methylbutan-1-ol

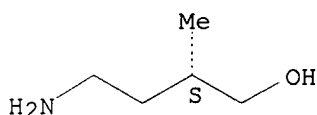
RL: IMF (Industrial manufacture); PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)

(recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resln.)

RN 44565-28-8 HCAPLUS

CN 1-Butanol, 4-amino-2-methyl-, (2S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



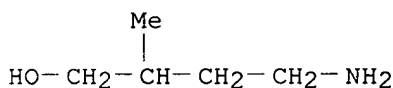
IT 44565-27-7, 4-Amino-2-methylbutan-1-ol

RL: RCT (Reactant); RACT (Reactant or reagent)

(recovery of **optically** active org. acid in prepn. of **optically** active 4-amino-2-methylbutan-1-ol by **optical** resln.)

RN 44565-27-7 HCAPLUS

CN 1-Butanol, 4-amino-2-methyl- (6CI, 9CI) (CA INDEX NAME)



L9 ANSWER 6 OF 9. HCAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2002:205034 HCAPLUS

DOCUMENT NUMBER: 136:247346

TITLE: Recovery of **optically** active organic acids and **optically** active amines in **optical** resolution

INVENTOR(S): Yamaguchi, Yasumasa; Hamanaka, Koji; Shibuya, Nozomu

PATENT ASSIGNEE(S): Mitsubishi Rayon Co., Ltd., Japan

SOURCE: Jpn. Kokai Tokkyo Koho, 6 pp.

CODEN: JKXXAF

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

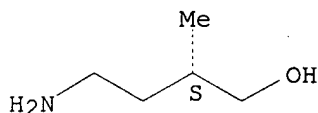
PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
JP 2002080407	A2	20020319	JP 2000-268507	20000905
PRIORITY APPLN. INFO.:			JP 2000-268507	20000905
OTHER SOURCE(S):		MARPAT 136:247346		

- AB Alc. solns. contg. title compds. are contacted by alkali carbonate and the pptd. **optically** active org. acid alkali salt crystals are sepd. by solid-liq. sepn. to recovery the **optically** active acids and amines. Thus, EtOH soln. contg. (R)-2-chloromandelic acid (I) and racemic 4-amino-2-methylbutan-1-ol was cooled, filtered, the filtrate refluxed with Na<sub>2</sub>CO<sub>3</sub> for 10 h, cooled, and filtered to give I salt and filtrate. The salt was dissolved in hot water, treated with HCl, and cooled to recover 87% I. The filtrate was distd. to give (S)-4-amino-2-methylbutan-1-ol with 61% e.e. with 88% recovery.
- IC ICM C07B063-00  
ICS C07C051-43; C07C059-56; C07C213-10; C07C215-08; C07B057-00;  
C07M007-00
- CC 23-7 (Aliphatic Compounds)  
Section cross-reference(s): 21
- ST **optically** active chloromandelic acid recovery aminomethylbutanol resoln; alkali carbonate **optically** active acid amine recovery; alc solvent **optical** resoln recovery; sodium carbonate **optically** active chloromandelic acid ethanol
- IT Carboxylic acids, preparation  
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(chiral; recovery of **optically** active org. acids and amines in **optical** resoln.)
- IT Sulfonic acids, preparation  
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(**optically** active; recovery of **optically** active org. acids and amines in **optical** resoln.)
- IT Resolution (separation)  
(recovery of **optically** active org. acids and amines in **optical** resoln.)
- IT Alcohols, uses  
RL: NUU (Other use, unclassified); USES (Uses)  
(recovery of **optically** active org. acids and amines in **optical** resoln.)
- IT Carbonates, reactions  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(recovery of **optically** active org. acids and amines in **optical** resoln.)
- IT **44565-28-8P**, (S)-4-Amino-2-methylbutan-1-ol **88390-32-3P**, (R)-4-Amino-2-methylbutan-1-ol  
RL: IMF (Industrial manufacture); PUR (Purification or recovery); SPN (Synthetic preparation); **PREP (Preparation)**  
(recovery of **optically** active org. acids and amines in **optical** resoln.)
- IT 52950-18-2P 328086-85-7P  
RL: IMF (Industrial manufacture); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
(recovery of **optically** active org. acids and amines in **optical** resoln.)
- IT 64-17-5, Ethanol, uses 67-56-1, Methanol, uses  
RL: NUU (Other use, unclassified); USES (Uses)  
(recovery of **optically** active org. acids and amines in **optical** resoln.)
- IT 497-19-8, Sodium carbonate, reactions **44565-27-7**, 4-Amino-2-methylbutan-1-ol  
RL: RCT (Reactant); RACT (Reactant or reagent)

(recovery of **optically** active org. acids and amines in  
**optical** resoln.)

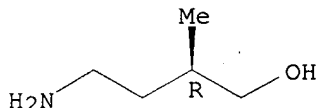
IT 44565-28-8P, (S)-4-Amino-2-methylbutan-1-ol 88390-32-3P,  
(R)-4-Amino-2-methylbutan-1-ol  
RL: IMF (Industrial manufacture); PUR (Purification or recovery); SPN  
(Synthetic preparation); **PREP (Preparation)**  
(recovery of **optically** active org. acids and amines in  
**optical** resoln.)  
RN 44565-28-8 HCAPLUS  
CN 1-Butanol, 4-amino-2-methyl-, (2S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

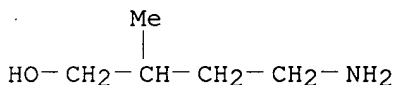


RN 88390-32-3 HCAPLUS  
CN 1-Butanol, 4-amino-2-methyl-, (2R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 44565-27-7, 4-Amino-2-methylbutan-1-ol  
RL: **RCT (Reactant); RACT (Reactant or reagent)**  
(recovery of **optically** active org. acids and amines in  
**optical** resoln.)  
RN 44565-27-7 HCAPLUS  
CN 1-Butanol, 4-amino-2-methyl- (6CI, 9CI) (CA INDEX NAME)



L9 ANSWER 7 OF 9 HCAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 2001:185708 HCAPLUS

DOCUMENT NUMBER: 134:193127

TITLE: Process for producing **optically active**  
aminoalcohol

INVENTOR(S): Ozaki, Eiji; Endou, Takakazu; Yamaguchi, Yasumasa;  
Hamanaka, Mitsuharu

PATENT ASSIGNEE(S): Mitsubishi Rayon Co., Ltd., Japan

SOURCE: PCT Int. Appl., 43 pp.

CODEN: PIXXD2

DOCUMENT TYPE: Patent

LANGUAGE: Japanese

FAMILY ACC. NUM. COUNT: 1

*n P Act*

## PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
WO 2001017944	A1	20010315	WO 2000-JP6092	20000907
W: JP, US				
RW: AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE				
EP 1219593	A1	20020703	EP 2000-956999	20000907
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI, CY				

PRIORITY APPLN. INFO.:

	JP 1999-252902	A	19990907
	JP 2000-205074	A	20000706
	WO 2000-JP6092	W	20000907

OTHER SOURCE(S): MARPAT 134:193127

AB Described is an industrially advantageous process for inexpensively producing **optically** active high-purity 4-amino-2-methylbutan-1-ol which is useful as an intermediate in synthesizing **optically** active medicines and pesticides. Racemic 4-amino-2-methylbutan-1-ol is treated with an **optically** active org. acid. The diastereomeric salt thus formed is crystd. out and subjected to solid-liq. sepn. to give **optically** active 4-amino-2-methylbutan-1-ol. This diastereomeric salt of **optically** active 4-amino-2-methylbutan-1-ol with an **optically** resolving agent is desalted by bringing into contact with a solvent and an alkali and subjecting to solid-liq. sepn., thereby recovering the **optically** active 4-amino-2-methylbutan-1-ol from the filtrate. Further, the filtration residue contg. the alkali salt of the **optical** resolving agent obtained by the solid-liq. sepn. is brought into contact with a solvent and an acid. Then the **optical** resolving agent thus crystd. out is subjected to solid-liq. sepn. and recovered. Thus, 110 g racemic 4-amino-2-methylbutan-1-ol (I) was added dropwise to a soln. of 200 g (R)-2-chloromandelic acid (II) in 1,100 mL isopropanol, heated to dissoln., cooled to room temp., treated with 100 mg (R)-I.II salt seed crystals, and further cooled slowly to 5.degree.. The pptd. crystals were sepd. by filtration and rinsed with cold isopropanol to give 173 g (R)-I.II salt (63 %e.e.) which was recrystd. from 1,100 mL isopropanol to give 102 g (R)-I.II salt (99.9 %e.e.). The latter diastereomer salt (100 g) was dissolved in 415 g MeOH upon warming, treated dropwise with 28% NaOMe/MeOH (80 g), cooled to 5.degree., subjected to solid-liq. sepn. The sepd. liq. was concd. and vacuum-distd. to give 15 g (R)-I (99.9 %e.e.). The sepd. solid was dissolved in 80 g water upon heating, treated with 7 g concd. H2SO4, cooled to 5.degree., and subjected to solid-liq. sepn. to recover 22 g II.

IC ICM C07C213-10  
ICS C07C215-08; C07B057-00

CC 23-7 (Aliphatic Compounds)

ST **optically** active aminoalc prepn; aminomethylbutanol  
**optical** resoln mandelic acid

IT Alcohols, preparation  
RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)  
(amino; prepn. of **optically** active aminoalc. by **optical** resoln. involving preferential crystn. of diastereomer salts with **optically** active acids and salt decompn.)

IT Carboxylic acids, reactions  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(chiral; prepn. of **optically** active aminoalc. by

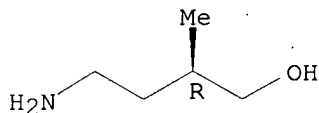
- optical** resoln. involving preferential crystn. of diastereomer salts with **optically** active acids and salt decompn.)
- IT Sulfonic acids, reactions  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (**optically** active; prepn. of **optically** active aminoalc. by **optical** resoln. involving preferential crystn. of diastereomer salts with **optically** active acids and salt decompn.)
- IT Resolution (separation)  
 (prepn. of **optically** active aminoalc. by **optical** resoln. involving preferential crystn. of diastereomer salts with **optically** active acids and salt decompn.)
- IT 64-17-5, Ethanol, uses 67-56-1, Methanol, uses 67-63-0, Isopropanol, uses 67-64-1, Acetone, uses 71-23-8, n-Propanol, uses 75-05-8, Acetonitrile, uses 108-20-3, Isopropyl ether 7732-18-5, Water, uses RL: NUU (Other use, unclassified); USES (Uses)  
 (crystn. solvent; prepn. of **optically** active aminoalc. by **optical** resoln. involving preferential crystn. of diastereomer salts with **optically** active acids and salt decompn.)
- IT 88390-32-3P, (R)-4-Amino-2-methylbutan-1-ol  
 RL: PUR (Purification or recovery); RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (prepn. of **optically** active aminoalc. by **optical** resoln. involving preferential crystn. of diastereomer salts with **optically** active acids and salt decompn.)
- IT 44565-28-8P, (S)-4-Amino-2-methylbutan-1-ol  
 RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP (Preparation)  
 (prepn. of **optically** active aminoalc. by **optical** resoln. involving preferential crystn. of diastereomer salts with **optically** active acids and salt decompn.)
- IT 98-88-4, Benzoyl chloride 124-41-4, Sodium methoxide 611-71-2, (R)-Mandelic acid 1310-73-2, Sodium hydroxide 2743-38-6, (-)-Dibenzoyl-L-tartaric acid 3144-16-9, (+)-10-Camphorsulfonic acid 17916-88-0, N-Acetyl-D-valine 20312-36-1, (S)-(-)-3-Phenyllactic acid 32189-36-9, (R)-4-Chloromandelic acid 44565-27-7, 4-Amino-2-methylbutan-1-ol 52950-18-2, (R)-2-Chloromandelic acid 52950-19-3, (S)-2-Chloromandelic acid 77977-73-2, (R)-4-Nitromandelic acid  
 RL: RCT (Reactant); RACT (Reactant or reagent)  
 (prepn. of **optically** active aminoalc. by **optical** resoln. involving preferential crystn. of diastereomer salts with **optically** active acids and salt decompn.)
- IT 328086-85-7P 328086-87-9P 328086-88-0P 328086-89-1P  
 RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent)  
 (prepn. of **optically** active aminoalc. by **optical** resoln. involving preferential crystn. of diastereomer salts with **optically** active acids and salt decompn.)
- IT 328086-86-8P 328086-90-4P 328086-91-5P 328086-92-6P 328086-93-7P 328086-94-8P  
 RL: SPN (Synthetic preparation); PREP (Preparation)  
 (prepn. of **optically** active aminoalc. by **optical** resoln. involving preferential crystn. of diastereomer salts with **optically** active acids and salt decompn.)
- IT 88390-32-3P, (R)-4-Amino-2-methylbutan-1-ol

RL: PUR (Purification or recovery); RCT (Reactant); SPN  
(Synthetic preparation); PREP (Preparation); RACT (Reactant  
or reagent)  
(prepn. of **optically** active aminoalc. by **optical**  
resoln. involving preferential crystn. of diastereomer salts with  
**optically** active acids and salt decompn.)

RN 88390-32-3 HCAPLUS

CN 1-Butanol, 4-amino-2-methyl-, (2R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



IT 44565-28-8P, (S)-4-Amino-2-methylbutan-1-ol

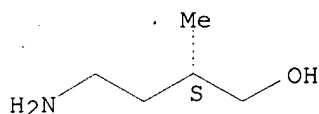
RL: PUR (Purification or recovery); SPN (Synthetic preparation); PREP  
(Preparation)

(prepn. of **optically** active aminoalc. by **optical**  
resoln. involving preferential crystn. of diastereomer salts with  
**optically** active acids and salt decompn.)

RN 44565-28-8 HCAPLUS

CN 1-Butanol, 4-amino-2-methyl-, (2S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



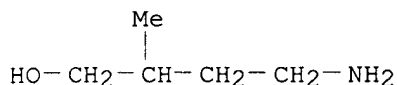
IT 44565-27-7, 4-Amino-2-methylbutan-1-ol

RL: RCT (Reactant); RACT (Reactant or reagent)

(prepn. of **optically** active aminoalc. by **optical**  
resoln. involving preferential crystn. of diastereomer salts with  
**optically** active acids and salt decompn.)

RN 44565-27-7 HCAPLUS

CN 1-Butanol, 4-amino-2-methyl- (6CI, 9CI) (CA INDEX NAME)



REFERENCE COUNT: 13 THERE ARE 13 CITED REFERENCES AVAILABLE FOR THIS  
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L9 ANSWER 8 OF 9 HCAPLUS COPYRIGHT 2003 ACS

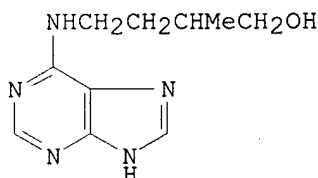
ACCESSION NUMBER: 1984:34316 HCAPLUS

DOCUMENT NUMBER: 100:34316

TITLE: Dihydrozeatin: an improved synthesis and resolution  
of both isomers

AUTHOR(S): Corse, J.; Gaffield, W.; Lundin, R. E.

CORPORATE SOURCE: West. Reg. Res. Cent., U.S. Dep. Agric., Berkeley, CA, 94710, USA  
 SOURCE: Journal of Plant Growth Regulation (1983), 2(1), 47-57  
 CODEN: JPGRDI; ISSN: 0721-7595  
 DOCUMENT TYPE: Journal  
 LANGUAGE: English  
 GI



AB (.-.-)-Dihydrozeatin (I) was synthesized by a Michael condensation of  $\text{CH}_2:\text{CMeCO}_2\text{Me}$  with  $\text{MeNO}_2$  to give (.-.-)- $\text{O}_2\text{NCH}_2\text{CH}_2\text{CHMeCO}_2\text{Me}$ , which was reduced to (.-.-)- $\text{H}_2\text{NCH}_2\text{CH}_2\text{CHMeCH}_2\text{OH}$  and treated with 6-chloropurine. Hydrolysis of the racemic ester gave (.-.-)- $\text{O}_2\text{NCH}_2\text{CH}_2\text{CHMeCO}_2\text{H}$ , which was resolved by means of (+)- and (-)-.alpha.-methylbenzylamine salts. Conversion of the salts to the corresponding Me esters and subsequent redns. yielded **optically** active  $\text{H}_2\text{NCH}_2\text{CH}_2\text{CHMeCH}_2\text{OH}$ . The sp. rotations at 589 nm of (S)-(-)- and (R)-(+)-I derived from **optically** active butanols were appreciably lower than previously reported. The biol. activities of (R)-, (S)-, and (R,S)-I in the betacyanin stimulation assay with *Amaranthus* parallel the activities found in other cytokinin bioassays.

CC 26-9 (Biomolecules and Their Synthetic Analogs)

IT 64070-19-5P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (prepn. and acylation of)

IT 44565-28-8P 88390-32-3P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (prepn. and reaction of, with chloropurine)

IT 64070-19-5P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (prepn. and acylation of)

RN 64070-19-5 HCAPLUS

IT 44565-28-8P 88390-32-3P

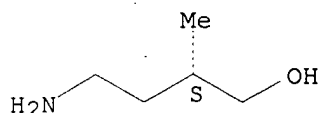
RL: RCT (Reactant); SPN (Synthetic preparation); PREP (Preparation); RACT (Reactant or reagent) (prepn. and reaction of, with chloropurine)

RN 44565-28-8 HCAPLUS

CN 1-Butanol, 4-amino-2-methyl-, (2S)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.

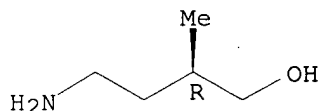
*not given by ref*  
*intermediate*  
*derivative*  
*isolated*



RN 88390-32-3 HCAPLUS

CN 1-Butanol, 4-amino-2-methyl-, (2R)- (9CI) (CA INDEX NAME)

Absolute stereochemistry.



L9 ANSWER 9 OF 9 HCAPLUS COPYRIGHT 2003 ACS

ACCESSION NUMBER: 1981:103753 HCAPLUS

DOCUMENT NUMBER: 94:103753

TITLE: An efficient synthesis of deuterated  
(.+-.)-dihydrozeatin-d5 and its riboside  
AUTHOR(S): Sugiyama, Tamizi; Tateba, Hideki; Hashizume, Takeshi  
CORPORATE SOURCE: Lab. Bioorg. Chem., Tokyo Inst. Agric. Technol.,  
Fuchu, 183, Japan

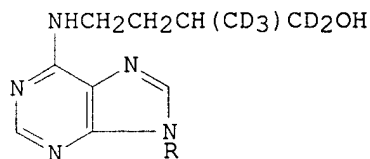
SOURCE: Agricultural and Biological Chemistry (1980), 44(11),  
2755-6

CODEN: ABCHA6; ISSN: 0002-1369

DOCUMENT TYPE: Journal

LANGUAGE: English

GI



AB (.+-.)-H<sub>2</sub>NCH<sub>2</sub>CH<sub>2</sub>CH(CD<sub>3</sub>)CD<sub>2</sub>OH, prepd. in 4 steps from .gamma.-  
butyrolactone, was condensed with 6-chloropurine or 6-chloro-9-.beta.-D-  
ribofuranosylpurine to give dihydrozeatins I (R = H, ribosyl) with 97.82  
and 97.23% deuteration.

CC 33-7 (Carbohydrates)

Section cross-reference(s): 28

IT 76742-83-1P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP  
(Preparation); RACT (Reactant or reagent)  
(prepn. and reaction of, chloropurines)

IT 76742-83-1P

RL: RCT (Reactant); SPN (Synthetic preparation); PREP

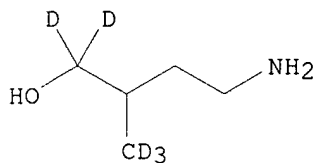
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(Preparation); RACT (Reactant or reagent)  
(prepn. and reaction of, chloropurines)

RN 76742-83-1, HCAPLUS

CN 1-Butan-1,1-d2-ol, 4-amino-2-(methyl-d3)- (9CI) (CA INDEX NAME)



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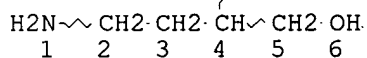
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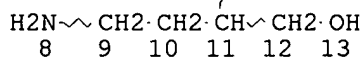
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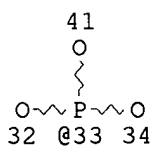
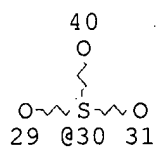
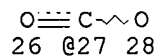
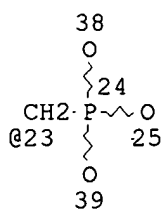
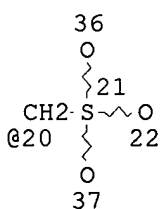
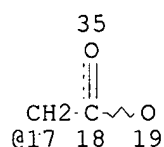
RRT

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Me



RRT

$$\begin{array}{c} \text{C} \sim \text{G1} \\ 15 \quad 16 \end{array}$$


VAR G1=17/20/23/27/30/33

NODE ATTRIBUTES:

CONNECT IS E1 RC AT 19  
 CONNECT IS E1 RC AT 22  
 CONNECT IS E1 RC AT 25  
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 CONNECT IS E1 RC AT 41

DEFAULT MLEVEL IS ATOM

DEFAULT ECLEVEL IS LIMITED

GRAPH ATTRIBUTES:

RING(S) ARE ISOLATED OR EMBEDDED

NUMBER OF NODES IS 41

STEREO ATTRIBUTES: NONE

L12: 0 SEA FILE=CASREACT SSS FUL L10 ( 0 REACTIONS)